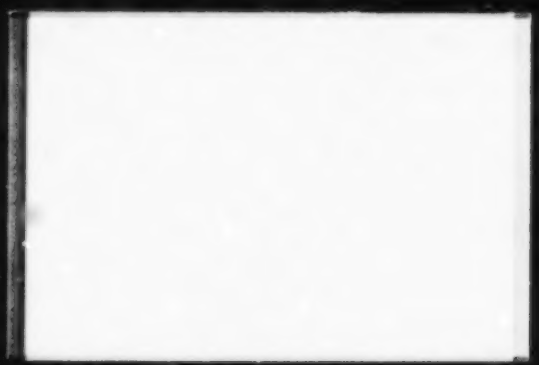

# RESEARCH

PROJECT REPORT

# AGGLOMERATION AND LIQUEFACTION OF HAT CREEK COAL

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and
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## 1.0 SUMMARY

A test program was conducted to investigate cleaning of Hat Creek Coal by oil agglomeration methods using (near) equilibrium recycle donor solvent as the agglomerating oil; deashed product was liquefied under previously established conditions.

Results of agglomeration testwork conducted by the Alberta Research Council (ARC) indicate that ground coal (median particle size 120 μ) can - after desliming - be agglomerated with recycle oil to produce coal with ash content as low as 13.3% (dry basis), which is equivalent to removal of 64.5% of the ash content of raw coal. Losses of coal to tailing from the cleaning process are mainly due to fine coal discarded in the desliming stage; for low ash coal (13.3-14.2%), recovery of calorific value to product was in the range of 63-65%. While this is not a high recovery, it compares favourably with conventional cleaning which produces a 21-22% ash product at 62-65% recovery. Agglomeration test results also indicated that liquefaction recycle oil (with a proprietary surfactant) is a satisfactory bridging oil. Thus cleaning of coal for liquefaction could be done with process derived materials.

Results of donor solvent liquefaction tests on agglomerate products indicated that total coal conversion (to gas and THF solubles) is not significantly changed by ash removal; control tests on raw coal gave - within experimental precision - the same conversion as agglomerate by either gravimetric or calorific value measurement. Batch test results indicate up to 94.3% total conversion of coal with use of iron sulphide catalyst.

A single test on coal agglomerate in a bench scale continuous unit produced a total conversion (by calorific value) of 88.6%. This is slightly lower than the range (90-92%) of total conversion obtained in previous recycle tests, but the difference is minor and partially attributable to variation in feedrate and residence time due to malfunction of the feed pump.

Overall, results of liquefaction tests on low ash agglomerates are considered to indicate that the high liquefaction reactivity of Hat Creek coal is not related to its ash content. Thus reduction of ash in feed coal is desirable to simplify process unit operations, particularly liquid/residue separation, and to increase oil yield by reducing oil required to discharge the liquefaction residue.

## 2.0 INTRODUCTION

### 2.1 Background

The Hat Creek coal deposit is an extremely large fossil fuel resource. Previous B.C. Research studies have shown that conversion of (low grade) Hat Creek coal to liquid and gaseous fuels is a technically feasible option for exploitation of this resource. Testing in both batch and continuous liquefaction systems (1,2) has shown that >90% of the calorific value of raw coal can be converted to saleable liquid and gas fuels by direct liquefaction technology using donor solvent. To date, liquefaction testwork has been done on either run of mine coal or partially beneficiated coal produced by the catalyst addition technique used. Dry basis coal ash contents in the range of 25-40% have been tested. These high ash levels present a significant barrier to commercial operation of liquefaction systems, primarily in the product liquid/residue separation operation.

Conventional methods of coal cleaning are only moderately effective for Hat Creek coal. Results of an EPRI pilot plant program indicated the possibility of reducing ash to 21% (dry basis) for coarse coal (+0.5 mm) and to ~26% for 0.5 mm x 100 mesh fines (3).

Recent work at the Alberta Research Council and elsewhere has shown that the spherical agglomeration process, previously thought to be applicable only to bituminous rank coals, can be adapted to sub-bituminous and lignite coals by using specific combinations of heavy binding oil, light oil diluent, and surfactants. A sample of 30% ash Hat Creek coal was shipped to the Alberta Research Council for exploratory testing of agglomeration. Initial results demonstrated ash reduction of 50% on an oil and moisture free basis with high apparent calorific recoveries. No optimization of agglomeration test conditions was attempted in this initial program, so there appeared to be potential for further reduction in ash content. However, even the preliminary results indicated significantly lower coal ash content

(15%) than the best levels (21-26%) obtainable with conventional coal cleaning technology.

In the long term, Japan represents both a market and a source of capital for synthetic liquid fuel developments based on the Hat Creek coal resource. Japanese (and other) liquefaction technologies require low ($\leq$10%) coal ash contents to facilitate product/residue separation. To date, Japanese interest in the Hat Creek coal resource for liquefaction has been minimal due to its high ash content.

In view of promising initial agglomeration results, a joint B.C. Research - ARC project was proposed to investigate agglomeration of Hat Creek coal and liquefaction of the deashed product. Funding for the joint project was obtained from CANMET (50%), B.C. Hydro (25%) and the B.C. Ministry of Energy, Mines and Petroleum Resources (25%).

## 2.2 Objectives

As stated in the project proposal, objectives were:

- optimization of agglomeration parameters to determine the maximum limits of ash rejection and recovery of calorific value.

- determination of the extent to which light and heavy petroleum oils used in preliminary agglomeration tests can be substituted by recycle liquefaction donor solvent.

- confirmation by batch and continuous liquefaction tests that agglomerated coal retains its high liquefaction reactivity after removal of the maximum possible amount of mineral matter.

## 3.0 MATERIALS AND METHODS

### 3.1 Coal Sample

A sample of Hat Creek coal was obtained from a site stockpile. This material (designated Trench B) was crushed to ~2 mm top size and riffle split to provide subsamples for agglomeration and (control) liquefaction tests on raw coal.

### 3.2 Recycle Solvent

Hydrotreated donor solvent from previous locked cycle liquefaction tests on Hat Creek coal was mixed and split to provide a subsample (~3.5 L) for ARC agglomeration testwork. The remainder was reserved for use in liquefaction tests on raw and agglomerated coal.

### 3.3 Agglomeration Testing

Procedures used in ARC agglomeration testwork are outlined in the draft report of this work which is presented as Appendix 1.

### 3.4 Liquefaction

Liquefaction tests were done in batch ("tubing bomb") and continuous 1 L/h bench systems. Equipment and procedures were described in a previous reports (1, 2).

The only significant modification to previously reported procedures was the mode of iron (sulphide) catalyst addition; iron was added as reagent grade ferric oxide (100% <10 μ particle size). Carbon disulphide (1.5 x stoichiometric) was the source of sulphur, as in previous work.

## 4.0 RESULTS AND DISCUSSION

### 4.1 Coal Agglomeration

Agglomeration test data are presented in Appendix 1.

Due to the high clay content of raw coal, desliming (i.e., removal of fines) was found necessary to obtain satisfactory agglomeration.

Combustible losses of 19-48% (Appendix 1, Table 2) at progressively finer feed-sizes indicate that the significant losses of combustible material are mainly due to loss of fines in desliming. Losses of this magnitude would have a significant effect on cost of clean coal, so development of a modified agglomeration process capable of recovering fine coal in the presence of large amounts of clay would be highly desirable.

Results of two-stage agglomeration with interstage wet grinding (Appendix 1, Table 5) indicate a modest reduction in ash content from the 16-18% range in single stage to 13-14% with two stage cleaning.

Other interesting aspects of these data are that recycle oil bridging liquid at the lower of two addition levels produced the lowest reported ash content (13.3% for two stage agglomeration) of any test, and that variation of total oil addition from 5.6% to 12.1% appears to have a negligible effect on product ash content.

Reported addition level of the proprietary surfactant was 0.35% of raw coal. Cost of this material is not reported.

Results of proximate analyses on the two agglomerate samples forwarded to B.C. Research for liquefaction testing are presented in Table 1.

The sample with high bridging oil content (Agglomerate #1) and containing bitumen had a low (4.8%) moisture content and low (12.6%) ash content, reflecting the high oil content.

Agglomerate #2, prepared with 3% recycle solvent bridging oil, contained substantially more moisture (14.9%) than the sample (Agglomerate #1) prepared with 12% of 60:40 recycle oil/bitumen. The ash content of 14.5% (dry, oil free) in Agglomerate #2 was not substantially higher than that of Agglomerate #1, which suggests that recycle oil without bitumen addition offers comparable performance on ash removal to oil-bitumen mixtures.

Moisture contents of agglomerate products are not reported by ARC, so effects (if any) of higher recycle oil bridging liquid additive on product moisture cannot be assessed.

Reported agglomeration contact times (per stage, Appendix 1) were 7 minutes; subsequent flotation times were not reported, but are assumed to be of the same order of magnitude. Such contact times are comparable to requirements for microagglomeration/flotation of bituminous coals.

Overall, the data indicate that Hat Creek coal can be cleaned to ash contents in the range of 13-16% using low levels of oil and surfactant (3.5 and 0.35% respectively vs raw coal), and that process time requirements are of the same order of magnitude as for equivalent treatment of bituminous coals.

The major indicated problems are combustible losses of 20-40% (mainly associated with slime loss), and inability of the technology - to date - to obtain an ash content $\leq$10% as required for the "NEDOL" process.

## 4.2 Coal Liquefaction Tests

Results of batch and continuous liquefaction tests on Hat Creek agglomerates are presented in Tables 2 and 3.

All batch tests except raw coal controls were done on the coal-oil-bitumen agglomerate sample containing 12.6% ash (dry basis).

Initial tests (#1 and #2, Table 2) in duplicate on raw coal and agglomerate gave equivalent conversion on a calorific value basis (respective averages 87.4% and 85.9%) within experimental precision. Conversion calculated from weight loss measurement was higher for the agglomerate, due to its initial bridging oil content.

Further tests with iron catalyst (2% Fe vs dry, ash free coal) resulted in conversion of 90.0-94.7% of calorific value conversion for agglomerates.

Tests 4 and 5 (agglomerate and raw coal, maximum pressure 13.5 MPa, with catalyst) gave conversions for both samples in the 93.5-94.7% range. These are equivalent to the best results previously obtained with Hat Creek coal (2)), and do not indicate any difference in performance between low ash agglomerate and high ash raw coal.

Also, use of fine iron oxide as the added form of iron in these tests (compared to previous results obtained with ferrous sulphate ion exchange treatment (2)), indicates that the ion exchange procedure is not necessary to obtain adequate catalyst dispersion.

Total conversion from the continuous test on oil agglomerated coal was 88.6% (Table 3). This value is at the low end of the range (89-93%) of conversion obtained in previous solvent recycle tests under nominally equivalent conditions. Part of the (slight) difference in conversion is attributed to unsteady performance of the autoclave feed pump during the test. The average retention time of 55 minutes was close to the target of 60 minutes, but periods of high pump flow resulted in short circuiting of feed through the single stage, back mixed reactor.

Byproduct gas yield (Table 3) was essentially the same as for a typical previous run on raw coal.

Liquid product quality, as indicated by toluene insoluble content (12%) of THF extract was significantly better than the average value of 18% toluene insolubles for previous solvent recycle tests, but within the range (11.8-22%) of previously measured values. To whatever extent the low toluene insoluble product is valid, it may indicate a slight improvement in raw liquid product quality.

In overall assessment of raw vs agglomerate coal reactivity, the consistency of the tubing bomb data is considered an important factor. These data indicate no analytically significant difference in conversion under a range of test conditions, with or without added catalyst.

On this basis, experimental evidence is considered adequate to conclude that liquefaction reactivity of Hat Creek coal is not affected by removal of 60% of its ash content. By inference, further beneficiation to ash levels of $\leq$10% would not affect its reactivity.

## 4.3 Canada-Japan Science and Technology Exchange

Papers (see Appendix 2) describing the Hat Creek coal deposit in general terms and the work on agglomeration/liquefaction test results were presented to a joint meeting in Tokyo on October 1, 1986.

The work was received with interest and a preliminary request for an agglomerate sample for testing was subsequently received.

The view of the high combustible losses in current testwork, and particularly the fact that the best agglomerate obtained to date contains 12.5% ash, it would be desirable to conduct further agglomeration testwork before submitting a sample for testing under NEDOL process conditions. A 10% ash content is considered by NEDOL to be the upper limit of acceptability for coal feedstocks. To submit a sample of material containing >10% ash would serve no useful purpose;

oil agglomerated coal is likely to be regarded as representing the (near) ultimate limit of physical cleaning technology. If such a sample exceeds the nominal ash specification of 10% (moisture free basis) for the NEDOL process, this may lead to the conclusion that the coal cannot be physically cleaned to the required ash content and is therefore not an acceptable fuel under any circumstances.

# 5.0 CONCLUSIONS

## 5.1 Agglomeration

With respect to cleaning of Hat Creek coal by oil agglomeration, test results are considered to support the following conclusions:

- Hat Creek coal can be cleaned to ~13% ash content (moisture free basis) using recycle solvent at 3.5% vs raw coal (dry basis).

- Use of donor solvent (with surfactant) produces agglomerates with ash content equivalent to or better than material produced using bitumen based bridging oil.

- Agglomeration by current test methods does not recover coal from the fine (<44 μ) fraction of raw coal.

- Use of agglomeration methods in the present state of development methods requires rejection of the slime (<44 μ) fraction of coal feed, resulting in a 35-40% loss of raw coal calorific value.

Rejection of 35-40% of raw coal calorific value as an aqueous slurry would significantly affect net mining costs due to both loss of fuel value and slurry disposal costs. On this basis, and considering the ash content limit of 10% for the NEDOL conversion process, it appears that further research is required to determine whether (and at what recovery) Hat Creek coal can be cleaned to prepare an acceptable feedstock for processes in advanced stages of development.

## 5.2 Liquefaction Reactivity

With respect to liquefaction reactivity, test data indicate that removal of up to 65% of raw coal ash does not significantly affect total conversion in donor solvent liquefaction. Equivalent conversions of clean and raw coal were obtained with or without added catalyst under a range of test conditions giving 84-94% total conversion.

Yield and composition of gas byproduct is also not significantly affected by removal of up to 65% of raw coal ash.

By inference, further reduction of ash by physical cleaning would not significantly affect reactivity or product slate.

For use of Hat Creek coal as a liquefaction feedstock, the critical remaining question thus appears to be whether further development of agglomeration technology will allow cleaning to $\leq$10% ash at an economically acceptable recovery.

## 6.0 RECOMMENDATIONS

Further laboratory scale research/development is required to demonstrate cleaning of Hat Creek coal to $\leq$10% ash at economically acceptable recovery.

R. O. McElroy
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## 7.0 REFERENCES

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2. B.C. Research. Continuation of the Evaluation of Hat Creek Coal for Direct Liquefaction. Prepared for CANMET (DSS23SQ.4-9681), B.C. Hydro, and B.C. Ministry of Energy, Mines and Petroleum Resources, December 1985.

3. EPRI. Coal Cleaning Tests on Hat Creek Coal. Prepared for B.C. Hydro, December 1982.

Table 1: ANALYSES OF HAT CREEK COAL AND AGGLOMERATES

| Sample | Proximate Analyses (wt%) | | | | | Calorific Value |
|---|---|---|---|---|---|---|
| | As Tested | | | | Dry, Oil Free | MJ/kg (As Tested) |
| | $H_2O$ | VM | FC | Ash | Ash | |
| Trench B Coal | 20.5 | 27.6 | 23.3 | 28.0 | 35.2 | 13.9 |
| Agglomerate #1 (binder: 60% recycle solvent, 40% bitumen; 12% vs. daf coal) | 4.8 | 50.3 | 36.1 | 8.7 | 12.6 | 25.1 |
| Agglomerate #2 (binder: recycle solvent; 3% vs. daf coal) | 14.9 | 36.4 | 36.7 | 11.9 | 14.5 | 19.5 |

Table 2: CONDITIONS AND CONVERSION DATA FOR BATCH LIQUEFACTION OF HAT CREEK COAL AND AGGLOMERATES

| Feed | System | Time (min) | Temp (°C) | Pressure (MPa) | Catalyst | Conversion (%) | | $H_2$ Consumptio (g/100 g) |
|---|---|---|---|---|---|---|---|---|
| | | | | | | Wt | Calorific Value | |
| Hat Creek Trench B | Batch | 25 | 425 | 10.5 | None | 82.6<br>84.9 | 86.5<br>88.4 | 0.8<br>1.0 |
| Agglomerate No. 1 (coal-oil-bitumen) | Batch | 25 | 425 | 10.5 | None | 86.5<br>86.2 | 85.7<br>86.0 | 0.6<br>ND |
| Agglomerate No. 1 (coal-oil-bitumen) | Batch | 55 | 425 | 10.7 | FeS | 90.0<br>89.3 | 90.0<br>90.8 | ND<br>ND |
| Agglomerate No. 1 (coal-oil-bitumen) | Batch | 25 | 425 | 13.5 | FeS | 94.8<br>93.7 | 94.0<br>94.7 | 2.4<br>2.2 |
| Agglomerate No. 2 (coal-3% oil) | Contin-uous | 55 (avg) | 425 | 14 | FeS | ND | 88.6 | ND |

Table 3: CONTINUOUS LIQUEFACTION TEST ON HAT CREEK AGGLOMERATE #2

| | | | |
|---|---|---|---|
| Average Retention Time: | 55 min | | |
| Temperature: | 425°C (±2°) | | |
| Slurry Feed: | 32% coal solids<br>Tetralin: naphthalene (solvent) = 3.4:1<br>Catalyst 2% Fe as $Fe_2O_3$ vs daf coal,<br>$CS_2$ sulphiding agent | | |
| Conversion: | Total: | 88.6% (calorific value)<br>89.8% (gravimetric) | |
| | Gas: | $CO_2$ | 9.9 wt% vs daf coal |
| | | CO | 0.7 |
| | | $CH_4$ | 3.2 |
| | | $C_2H_6$ | 1.9 |
| | | $C_3Hg$ | 1.2 |
| | | $C_4$ | 0.6 |
| | | Total | 16.1 |
| | Liquid: | THF soluble 65.8 wt% vs daf coal | |
| | | THF soluble/benzene insoluble 7.9 wt% vs daf coal | |
| Total Material Recovery: | $\frac{\text{(slurry + gas out)}}{\text{(slurry + gas in)}} \times 100 = 98.2\%$ | | |

# APPENDIX 1

# AGGLOMERATION OF HAT CREEK COAL

Alberta Research Council

# Agglomeration of Hat Creek Coal

by
J. Janiak and A. Turak

Submitted to: B.C. Research
Contract No.: 602001

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# 1 Introduction

Hat Creek coal has ben considered to be very reactive and a suitable feedstock for direct liquefaction process[1]. The high ash content of the run-of-mine Hat Creek coal however poses serious problems in processing. Therefore beneficiation is considered to be a logical step before liquefaction. Conventional beneficiation was not successfull when applied to this coal[2][3], but preliminary tests in Alberta Research Council (ARC) showed that it was possible to reduce ash content of Hat Creek coal by more than 50% by oil agglomeration.

The agglomeration process selectively removes certain minerals from the coal and this may result in altering the liquefaction reactivity.In addition to mechanical difficulties in processing high ash coal, this possible alteration of liquefaction reactivity was the major interest of B.C. Research.

Furthermore, B.C. Research was also interested in replacing bitumen based binder, typical of the ARC process, with the liquid products obtained from liquefaction of Hat Creek coal.

## 2 Work Statement

A joint research program with B.C. Research was initiated and the objectives of the agglomeration work, to be carried out by ARC were as folows:

1. Optimizing agglomeration parameters to obtain maximum de-ashing and high carbon recoveries,

2. Investigating possibility of using recycle oil as binder in agglomeration,

3. Preparing bulk agglomerate sample for the liquefaction tests and verifying the small tank agglomeration test results in larger scale.

The following sections of this report summarizes the experimental technique and presents the results of laboratory testing on beneficiation of Hat Creek coal by oil agglomeration.

# 3 Samples

Two coal samples and two oil samples were supplied by B.C. Research for this investigation. The coal samples were from Hat Creek deposit labeled Trench-B and Trench-A, and the oil samples were labeled as +180° C and -180° C Mixed Recycle Oils. The size analysis of the coal samples showed that the coal was below 5 mm top size and had a mean particle size of 1.8 mm. The proximate analysis revealed that coal contained 19% moisture and 34% ash. The second Trench-B sample was found to contain slightly higher ash at 38%.

## 3.1 Sample Preparation

The coal samples were air dried by spreading on the floor in the lab for 24 hours before crushing and grinding. The contents of one container were crushed below 2 mm and were quartered and split to obtain 500 g samples that were individually sealed in plastic bags.

For each run in the small stirred tank a new bag of sample was dry ground by disc pulverizer in a closed-loop system with an appropriate sieve to obtain the desired top size.

# 4 Experimental

The experimental program for this project was planned in three stages in accordance with the work statement.

The first stage agglomeration tests were conducted in small stirred tanks of approximately 2 liter capacity for optimization of important operating parameters. The stirred vessels were equipped with a mixer composed of one radial impeller at the tip of the shaft and an axial impeller above it. The diameter of the impellers were limited to 1/3 of tank diameter and the clearance from the spherical bottom of the tank was set at 1/5 of impeller size. The wessels were were also equipped with four removable baffles.

The received coal samples were dry ground to the desired top size prior to agglomeration, unless indicated otherwise (for instance: tests investigating the effects of wet grinding).

As it will be discussed later, desliming of coal samples was necessary to achieve the desired de-ashing and size growth. Desliming was done by either screening or by elutriation.

After desliming, wet coal samples were placed in the agglomeration tank and water was added to obtain the desired solids concentration. At this point a surfactant (UBH)* was added to the coal water slurry, and while stirring, the binder oil was introduced to the slurry without any pre-treatment or emulsification. The end of oil addition was taken as the on set of agglomeration process and the baffles were removed when inversion was observed (formation of micro-agglomerates which is evident by sudden color

*Proprietary

change of the slurry) subsequently stirring was continued without baffles to provide low-shear mixing resulting in agglomerate growth.

After formation of the agglomerates separation of the product from the mineral matter slurry was achieved either by wet screening and washing on laboratory sieves or by froth flotation in a standard Denver Laboratory Flotation Machine when agglomerate size was not sufficiently large.

# 5 Results and Discussion

The small scale stirred tank tests were aimed at optimizing the process in terms of maximizing the ash removal while maintaining maximum recovery of the carbonaceous material. The high clay content[4] of the feed coal posed serious problems in direct agglomeration and this adverse effect of fine mineral matter was particularly pronounced when recycle oil was used as binder. Since the ultimate purpose was to beneficiate the coal using the recycle oil, the investigation was firstly concentrated on elimination of mineral fines.

## 5.1 Desliming of Feed Coal

A study of ash distribution in different size fractions of the agglomeration feed suggested possible reduction in fines by desliming without very high combustable losses. According to the ash distribution given in Table 1, for a sample that was dry ground to -0.6 mm top size, it would be possible to reject ∿13% of total ash by desliming at 45 microns. Table 2 summarizes the results of various alternative desliming procedures.

Figure 1 shows that combustable recoveries rapidly decreased for coal median sizes below 0.2 mm, but remained above 80% for optimum size of 0.2 mm with a reduction in ash from 38% to 24%. Figures 2 and 3 depict the effect of wet grinding on the median size and size distribution of feed material before desliming.

Various methods were used to deslime the coal and the results are pre-

Table 1: Ash Distribution in Hat Creek Coal* as a Function of Size Distribution

| Coal Fraction [mm] | Size Distribution [%] | Ash Content [%]M.F | Ash Distribution [%]M.F |
|---|---|---|---|
| TOTAL | 100.0 | 36.8 | 100.0 |
| 0.6 - 0.1 | 75.5 | 34.3 | 70.3 |
| 0.1 - 0.045 | 14.5 | 41.3 | 16.3 |
| - 0.045 | 10.0 | 49.2 | 13.4 |

* $d_{50}$ = 0.197mm

Table 2: Influence of Size Reduction on Desliming and Deashing of Hat Creek Coal*

| TEST NO | FEED SIZE [mm] | METHOD OF GRINDING | $D_{50}$ [mm] | YIELD | ASH CONTENT % M.F | RECOVERY OF COMBUSTIBLE MATERIAL % daf |
|---|---|---|---|---|---|---|
| Feed Coal | -0.6 | Dry | 0.31 | -- | 38.4 | ---- |
| J-166 | -2.0 | Dry | 0.69 | 65.3 | 24.1 | 80.4 |
| J-170 | -0.6 | Dry | 0.31 | 66.3 | 24.5 | 81.3 |
| J-167 $d_{50}$ | -0.6 +0.31 | Wet in Ball mill 10 min. | 0.14 | 66.1 | 26.3 | 79.1 |
| J-168 $d_{50}$ | -0.6 +0.31 | Wet in Ball mill 20 min. | 0.09 | 58.2 | 25.7 | 70.2 |
| J-169 $d_{50}$ | -0.6 +0.31 | Wet in Ball mill 30 min. | 0.08 | 50.4 | 23.3 | 62.7 |

* - 0.6mm, $D_{50}$ = 0.31mm

UBH = 0.35%

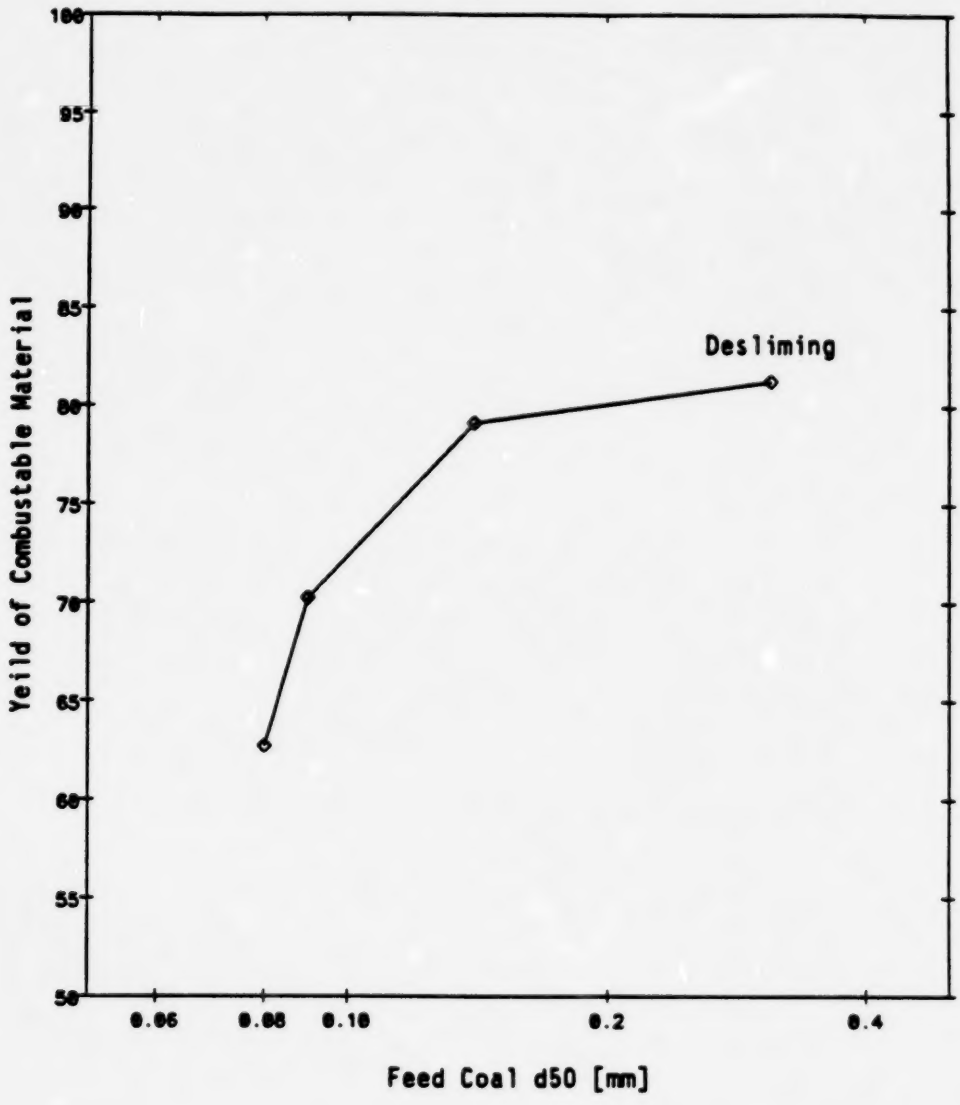


**Figure 1: Influence of Coal Size Reduction on Combustable Yield After the Desliming Process**

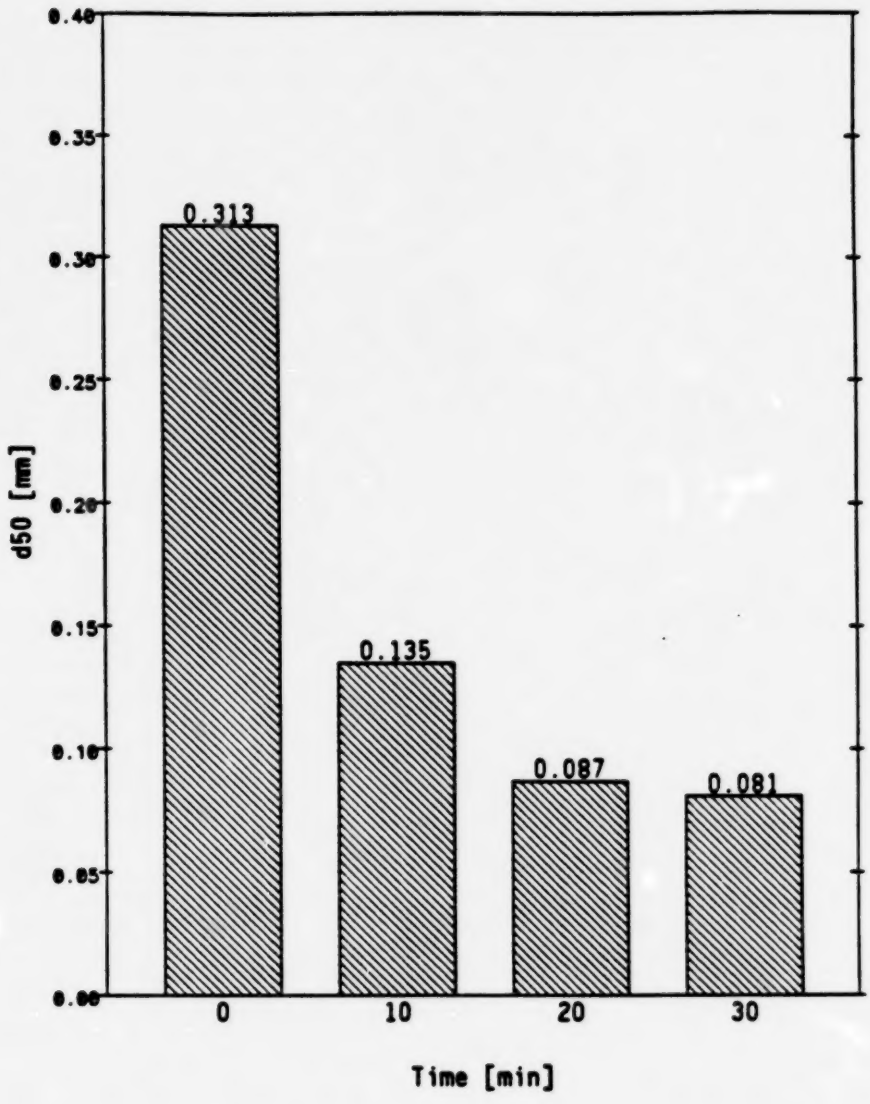


**Figure 2: Influence of Wet Grinding time on the Median Size of Hat Creek Coal**

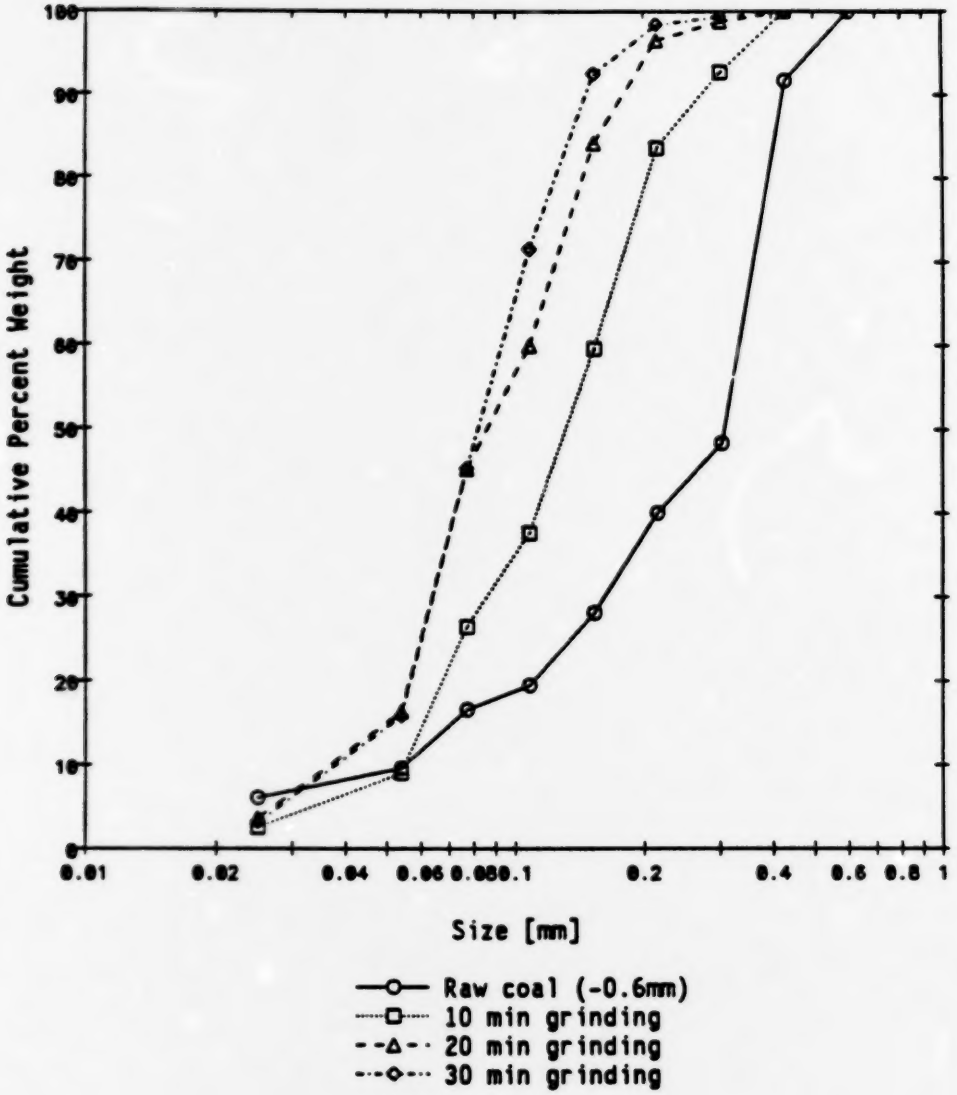


**Figure 3: Influence of Wet Grinding time on the Size Distribution of Hat Creek Coal**

sented in Table 3. Regardless of the method used the results were very much the same.

## 5.2 Agglomeration Test Results

Single stage agglomeration after wet grinding of deslimed feed and after subsequent separation by froth flotation revealed that wet grinding improved the recoveries and de-ashing slightly. Table 4 summerizes the results of tests on material ground in the ball mill for 10, 20 and 30 minutes. The overall yield of combustables for theses tests (after desliming-agglomeration-flotation) was about 60% while ash was reduced to 16.5% from 38 % by addition of 8% oil only.

Further ash reduction was possible by two stage agglomeration with interstage grinding. The froth from flotation of the first stage agglomeration product was reground in the ball mill for 10 minutes and was subsequently agglomerated by additional binder oil. Table 5 summerizes the results of optimized two stage agglomeration tests. By two stage agglomeration it was possible to reduce the ash content of agglomerates to 13% with 63-65% combustable recoveries.

Micrographs of the feed coal slurry, deslimed coal, separated fine minerals, agglomerated coal and the coarse reject from the single stage agglomeration process are presented in Appendix A.

Table 3: Influence of Different Methods of Desliming on Weight Loss and Deashing of Hat Creek Coal

Feed Top Size = 0.6mm/$D_{50}$ = 0.38mm

| DESLIMING METHOD | WEIGHT LOSS [%] M.F. | ASH CONTENT IN COAL [%] M.F. |
|---|---|---|
| Screening 0.3 mm | 29 | 20 |
| Elutriation in Column | 32 | 24 |
| Decantation | 27 | 21 |

Table 4: Influence of Particle Size on Deashing of Hat Creek Coal by Deliming-Agglomeration-Flotation

| TEST CONDITIONS | | | MICROAGGLOMERATES | | | |
|---|---|---|---|---|---|---|
| | OIL ADDITION | | | | | |
| $D_{50}$ [mm] | Dry Coal [%] | Time Of Agglomeration [min] | Yield of Combustible Material [%] | Ash Content [%] | Yield of Combustible Material [%] | Deashing [%] |
| 0.14 | 10.0 | 7 | 75.5 | 17.1 | 61.9 | 55.5 |
| 0.09 | 8.9 | 7 | 89.0 | 18.1 | 65.8 | 47.1 |
| 0.08 | 8.0 | 7 | 89.3 | 16.4 | 60.0 | 57.3 |

Table 5: Two Stage Agglomeration Results

CREEK COAL - Ash = 37.4% M.F  Bridging Liquid: B.C. Mixed Recycle Oil + 180°C  UBM - 0.35%

| | I. Stage AGGLOMERATION | | | | WET GRINDING | II. Stage AGGLOMERATION | | | OVER ALL BALANCE | |
|---|---|---|---|---|---|---|---|---|---|---|
| 50 mm] | OIL ADDITION M.F [%] | TIME AGGLOM-ERATION [min] | ASH CONTENT M.F [%] | DEASHING [%] | TIME [min] | TOTAL OIL ADDITION M.F [%] | TIME AGGLOMER-ATION [min] | ASH CONTENT M.F [%] | YIELD OF COMBUSTIBLE MATERIAL [%] | DEASHING [%] |
| .22 | 3.5 | 7 | 18.1 | 51.6 | 10 | 5.6 | 5 | 13.7 | 60.8 | 63.4 |
| | 10.0 | 7 | 15.6 | 58.3 | 10 | 12.1 | 5 | 14.2 | 63.3 | 62.0 |
| 0.12 | 3.5 | 7 | 14.9 | 60.2 | 10 | 5.6 | 5 | 13.3 | 66.4 | 64.5 |
| | 10.0 | 7 | 15.6 | 58.3 | 10 | 12.1 | 5 | 13.4 | 64.4 | 64.1 |

## 5.3 Large Tank Tests

The large tank tests were conducted at near-optimum conditions, and by one stage agglomeration it was possible to reduce the ash to 14-15%. The combustable recoveries were also comparable to the small tank test results at 62-66%. Table 6 summarizes properties of the products from the two tests carried out in a large tank and the reduction in ash and increase in calorific value for these two samples are depicted in Figures 4 and 5 respectively.

Table 6: Hat Creek Coal Agglomerate Samples from Large Tank Tests

| TESTS | COAL FEED | J-157 | J-161 |
|---|---|---|---|
| Ash Content % | 37.4 | 14.1 | 15.4 |
| Oil Addition to Agglomeration Process | ---- | 3.3 | 12.1 |
| Calorific Value BTU/LB | 7135 | 9796 | 9949 |
| Ash Removal % | ---- | 62.3 | 58.8 |
| Yield of Combustible Materials | ---- | 66 | 62 |

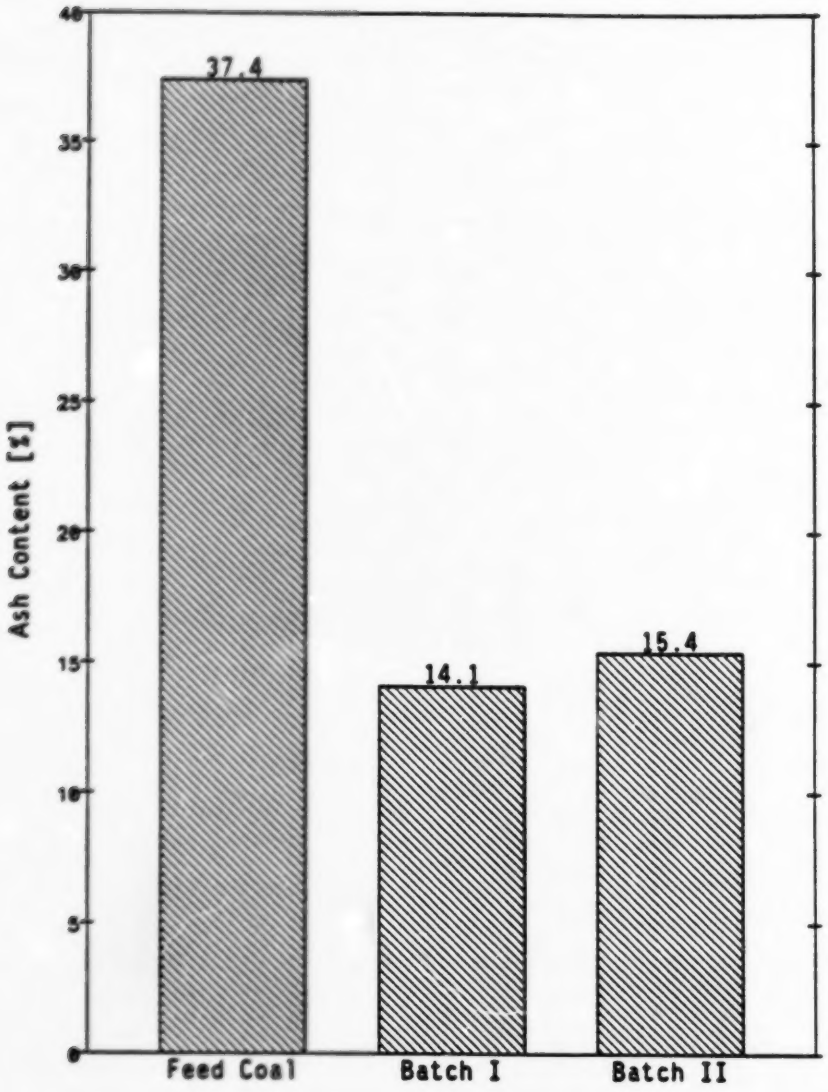


**Figure 4: Ash Content of Agglomerates from Large Tank Tests**

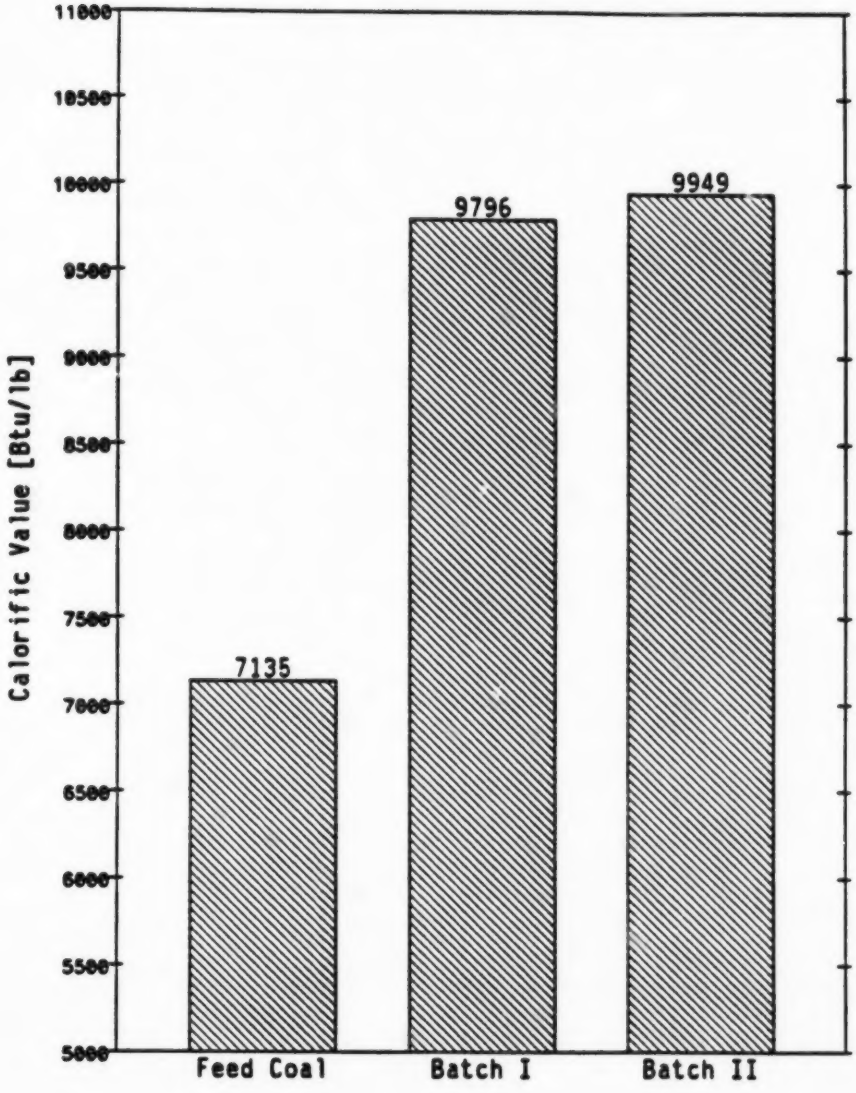


**Figure 5: Calorific Value of Agglomerates from Large Tank Tests**

# 6 Conclusions

The limited number of agglomeration tests conducted on Hat Creek coal in small stirred tanks revealed that:

- Trench-B coal samples containing 38% ash could be beneficiated to 15% ash levels using mixed recycle oil, obtained from liquefaction of this coal, as binder,
- Combustable recoveries in these optimized tests were over 65%,
- It was necessary to deslime the coal feed prior to agglomeration to facilitate sufficient agglomerate growth,
- Froth flotation was necessary to separate the micro-agglomerates after the first stage or in single stage agglomeration,
- Two stage agglomeration with interstage grinding was beneficial in further decreasing the ash content to 13% levels.

The results showed that oil agglomeration of Hat Creek coal is technically feasible and can be considered as a viable beneficiation method for this coal prior to its liquefaction.

The major shortcoming of the oil agglomeration method, as developed for this coal at this stage of investigations, is the low combustable recoveries. The total combustable losses for the optimized tests were in range of 30-35%. The agglomeration step recoveries for all tests were above 95%.

Most of the combustable losses occurred during desliming, prior to agglomeration. Losses of up to 35% are a major concern for mining economics.

Possible methods for reducing combustable losses include optimization of crushing and grinding, and/or identification of reagents (surfactants, oils, etc.) which will allow agglomeration of coal particles below 44 microns in size.

# 7 Recommendations

As it was indicated in the conclusions, the carbon losses of the three-stage (desliming-agglomeration-flotation) process are too high. Various alternative desliming and demineralisation techniques should be studied in order to improve carbon recoveries. The limited resources of this project did not permit an investigation of stagewise grinding and desliming. This could have a great effect on the agglomeration and separation steps of the process.

Another potential route to improving the carbon recoveries is through combining the desliming and microagglomeration steps by employing appropriate surface active reagents.

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[4] Woodley, R.M. 1983. Hat Creek Coal Utilization. B.C. Hydro and Power Authority. CIE/CIM Coal Symp. Calgary Alberta, May 18/19, 1983.

# Appendix

## A Micrographs of Feed and Agglomeration Products

Figure 6: Micrograph of Feed Coal Slurry

Figure 7: Micrograph of Deslimed Coal

Figure 8: Micrograph of Separated Fine Mineral Matter

Figure 9: Micrograph of Agglomerates

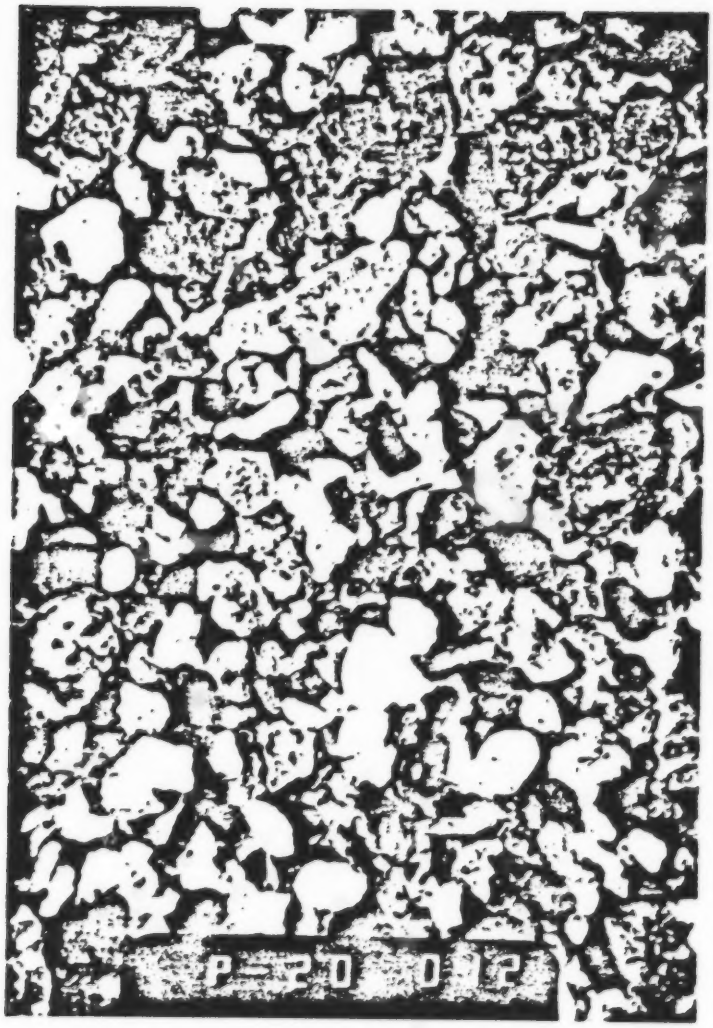

Figure 10: Micrograph of Coarse Reject